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CONTRIBUTIONS FROM THE CHEMICAL LABORATORY
OF HARVARD COLLEGE.

A NEW METHOD FOR THE DETERMINATION OF THE SPECIFIC HEATS OF LIQUIDS.

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During the course of an extended research upon heats of neutralization now in progress, it became necessary to devise some method for the accurate determination of the specific heats of the reacting solutions. Obviously an accurate value for any thermochemical measurement can only be obtained when the factors involved in the calculation are accurately ascertained; and it is well known that the existing data on this subject are by no means satisfactory. The recognized sources of error of the majority of the earlier methods and the discrepancies observed in the values obtained by the different experimenters using them[1] limit any dependence which can be placed in the constants thus obtained. Further, the truth of the assumptions upon which the corrections for their errors are based is by no means adequately proved. To obviate the necessity of these corrections, and thus eliminate the uncertainty attending their use, a new method has been devised. A brief discussion of the earlier forms of apparatus may assist in a better understanding of the difficulties encountered in devising this method and the means by which they were surmounted.

Of the various methods recorded, that of Andrews[2] has been, perhaps, the most frequently used. This depended upon the transference of a heated object or "calorifer" from a source of heat to the calorimeter, which contained either water or the liquid to be studied. A compar-

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[1] The following is a typical example:

Specific Heat of NaOH

%	Sp. Ht.	Observer.
49.5	0.816	Hammerl.
25.6	0 869	Hammerl.
22.9	0.847	Thomsen.

[2] Pogg. Ann., **75**, 335 (1848).

ison of the observed rise with water and with the liquid under investigation gave a simple means of determining the relative heat capacities. A variant of this method consisted in using either water or the studied liquid in the calorifer, the calorimeter always being filled with the former. This method, with various independent modifications, was used by Schüller,[3] Person,[4] Pfaundler,[5] Marignac,[6] Hammerl,[7] and a number of other investigators. The simplicity of this procedure, and the elimination of many doubtful factors by using comparative results, are strong arguments for its use; but the interchange of heat by radiation between both the calorifer and the calorimeter and their environments, coupled with the unavoidable lag of the thermometer, introduces elements of uncertainty fatal to the highest accuracy.

The ingenious device of Thomsen,[8] whereby measured amounts of hydrogen are burned, under constant pressure, inside the calorimetric system, gave concordant results; but the values obtained are subject to some of the same corrections as those demanded by the Andrews method. Pfaundler,[9] using electrical energy as his source of heat, attempted automatically to eliminate the radiation-correction by heating simultaneously two calorimeters, one containing water, the other the liquid under investigation. If the rise of temperature were the same, the loss by radiation would cancel. But as varying heat capacities involve varying amounts of electrical energy to secure this result, the electrical heat unit enters the computation, and by its uncertainty detracts from the absolute accuracy of the determination. This device has been recently applied in a modified form by Magie [10] with considerable success; but it is by no means easy to find a heat-producing electrical resistance suitable for immersion in electrolytes.

Several other different methods have been suggested by others, but these also are not wholly free from defect. In one, the radiation method of Dulong and Petit,[11] the hot object was enclosed in an evacuated and blackened chamber, losing its heat by radiation. The chamber was placed either in an ice bath or in a water bath of sufficient size to be unaffected by the heat given up by the cooling object. The relative temperatures of the hot object and its environment, and the time required to secure thermal equilibrium, gave the necessary data. The uncertainty of the true law of cooling is enough to seriously impair the accuracy of any results thus obtained, however.

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[3] Ann. de Chim. et Ph., **3**, 33, 437.

[4] Pogg. Ann., **136**, 70, 235 (1869).

[5] Wien. Ber., **62**, (2), 379 (1870).

[6] Arch. Gen., **2**, 39, 217 (1870); **2**, 55, 113 (1876).

[7] C. R., **90**, 694 (1880).

[8] Thermochem. Untersuch., **1**, 24 et seq. (1882); Pogg. Ann., **142**, 337 (1871).

[9] Wien. Ber., **59**, (2), 145 (1869); **100**, (2a), 352 (1891).

[10] Phys. Rev., **9**, 65 (1899); **13**, 91 (1901); **14**, 193 (1902); **17**, 105 (1903).

[11] Ann. de Chim. et Ph., **2**, 10, 395 (1819).

Quite a different procedure was adopted by Hesehus,[12] who balanced the heating effect of the calorifer in a calorimeter at room temperature by the additions of successive portions of cold water. In this way he eliminated any cooling of the calorimeter. Waterman[13] improved this method, and made a series of apparently excellent determinations of the specific heats of metals. Using a Pfaundler resistance coil as a source of heat, Litch[14] has studied in this way the specific heat of water. Satisfactory as these methods may appear upon first sight to be, however, the unavoidable warming of the cold water during its transference to the warm calorimeter introduces an element of uncertainty just as great as the uncertainty in the ordinary cooling correction; hence no real gain was made. The method is not really adiabatic.

In 1905 a new method was described by Richards and Lamb,[15] eliminating most of the earlier sources of error while maintaining all the advantages of the older procedure except simplicity. Two portions of liquid — one hot, the other cold — were rapidly discharged from their respective containers and mixed in a calorimeter, the temperature of the mixture being that of the environment. Obviously, the cooling experienced by the warm liquid during transference is balanced by the warming of the cold liquid. The method involves a somewhat high degree of mechanical complexity, and is further complicated by the necessity of making supplementary determinations of the heats of solution or dilution where the two liquids possess any degree of mutual solubility.

More recently a new method of calorimetry, by a strictly adiabatic procedure, has been described by Richards,[16] and its applicability has been experimentally proved by the same investigator with the assistance of Forbes,[17] Henderson,[18] and Frevert.[19] Here the *environment* of the calorimeter is caused to increase in temperature as the calorimeter itself becomes warmer. The studied transformation in the calorimeter thus takes place without interchange of heat with the surroundings. Further, since both the initial and the final temperatures are stationary, the error due to the lag of the thermometer disappears.

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[12] Jour. Soc. Ph. Chim. Russ., Nov., 1887; Jour. de Phys., **7**, 489 (1888).

[13] Phys. Rev., **4**, 161 (1896).

[14] Ibid., **5**, 182 (1897).

[15] These Proceedings, **40**, 659 (1905).

[16] Ibid., **41**, 8 (1905).

[17] Ibid., **41**, 10 (1905).

[18] Ibid., **41**, 10 (1905); **42**, 573 (1907).

[19] Ibid., **42**, 573 (1907).

The use of this method obviates at once the greatest source of error in calorimetric work of all kinds, namely, the correction for cooling. As the method may be employed in any kind of calorimetric work, there seemed to be no reason why it should not be applicable to work on specific heats; and the present paper will show that it is indeed of great service there. The application is extremely simple: the substance to be studied should obviously be placed in a calorimeter surrounded on all sides by a jacket, the temperature of which can be changed to correspond exactly with the warming of the substance by some known source of heat.

It was first necessary to decide upon the exactly quantitative source of energy to be used for heating the substance within the calorimeter. Some experimenters have used merely the heat of a warmer body; others have used electrical heat; and Thomsen availed himself of the heat of combustion of hydrogen. Of course many other chemical reactions might be employed for this purpose, as Ostwald and Luther have pointed out[20]; and after much consideration there was selected for this present work the heat of neutralization of pure sulphuric acid and sodic hydroxide as the most convenient, especially because it is not very changeable with the temperature.

Definite amounts of acid and alkali were allowed to react in a platinum flask surrounded by the liquid in the calorimeter, and the rise of temperature in the whole system was carefully noted. By comparing the rise of temperature under these conditions with the rise shown when pure water is in the calorimeter, a comparative measurement of the heat capacity of the liquid is made. A few words will suffice to explain the disposition of the apparatus and the method of its use.

APPARATUS.

A diagrammatic sketch of the apparatus in vertical section is seen in Figure 1. First, the environment of the calorimeter will be described. The jacket (*A*) was made of heavy sheet copper and was provided with an outflow cock (*V*) for convenience in emptying. The soldered joints were heavily coated with shellac to prevent corrosion by the alkaline solution with which it was filled. The capacity was 17.5 liters. A rotary, vaned stirrer (*R*), with a speed of 145 turns per minute, insured thermal homogeneity in the contents of the jacket. To raise the temperature, crude sulphuric acid was run into the jacket through the funnel (*F*), into the alkali contained in the jacket, and the heat of neutralization thus liberated was rapidly disseminated throughout

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[20] Ostwald-Luther, Phys. chem. Messungen (1902), p. 204.

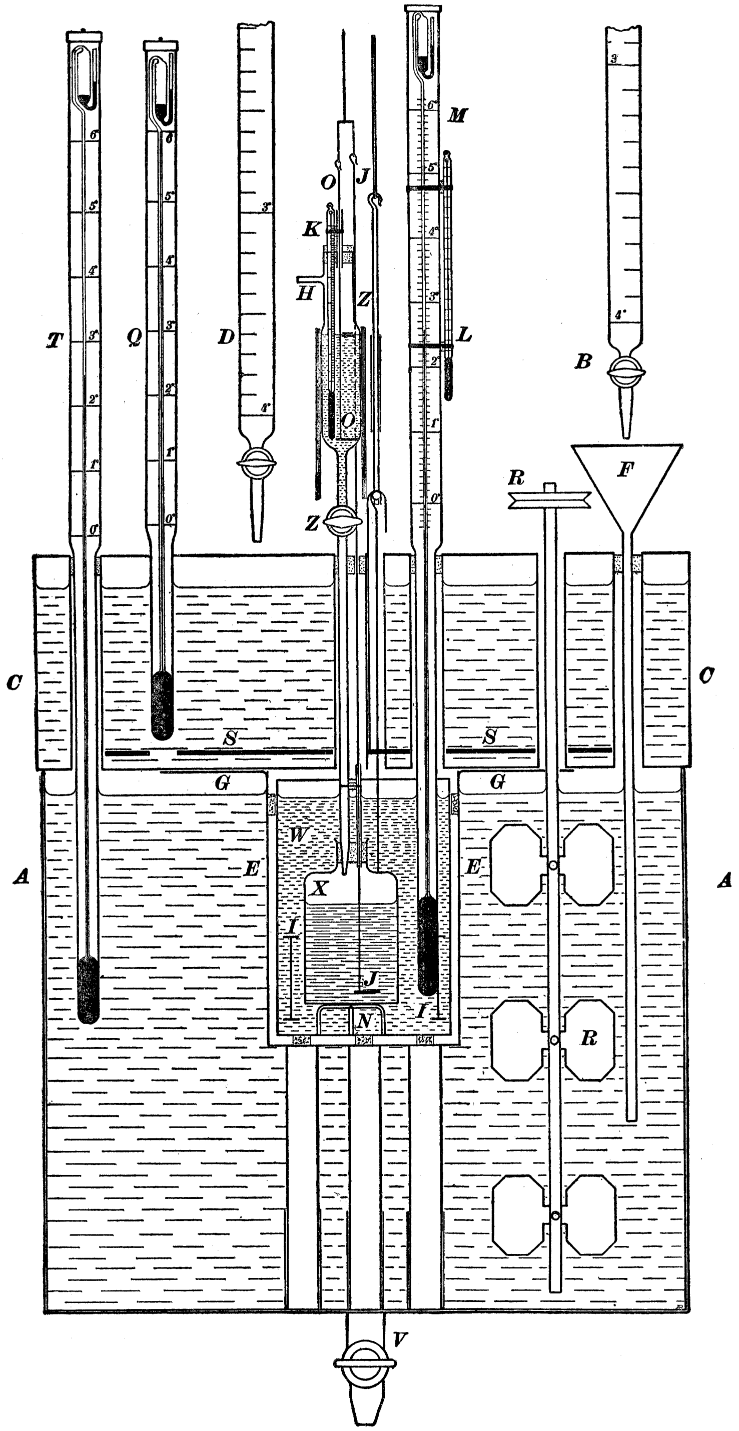

Figure 1.

the entire mass of liquid. The acid was contained in the burette (*B*) empirically graduated to give a rise of 0.1° for each scale division. The Beckmann thermometer (*T*), graduated in twentieths of a degree, indicated the temperature. The cover (*C*) was similarly constructed, the capacity being 6 liters. It was furnished with an oscillating stirrer (*S*) with a speed of 45 strokes per minute, and the Beckmann thermometer (*Q*) similar to that in the jacket. In the same way acid was admitted from the burette (*D*), suitably graduated. Copper tubes, permitting the passage of those portions of the apparatus which projected below the cover, were soldered to the bottom, and the joints were protected by a coating of shellac. The cover must fit tightly, otherwise evaporation will cause a slight cooling effect. The vessel was thoroughly cleaned at the end of each day's work. The inner cylinder (*E*) used to hold the calorimeter proper, was of sheet copper, nickel plated, and burnished on the inner surface. It was mounted on three legs, fitting into holders soldered to the bottom of the jacket, and was provided with the ring or apron (*G*), which prevented any portion of the liquid in the jacket from being thrown by the rapid stirring into its interior space.

Inside this inner cylinder and separated from it by points of dry cork was the calorimeter proper (*W*). This was a platinum can of 0.7 liter capacity, weighing 107 grams. During an experiment this was filled with water, or with the liquid the specific heat of which was to be measured. Thermal homogeneity of the calorimeter contents was secured by the two-stage perforated platinum stirrer (*I*) driven at a speed of 45 oscillations per minute. The temperature was accurately indicated by a large-bulbed, Beckmann thermometer (*M*), which was graduated in hundredths of a degree and capable of being read within $\frac{1}{1000}$. A small auxilliary thermometer (*L*) gave the temperature of the exposed stem. Thus far the apparatus is essentially similar to that used by Richards, Henderson, and Frevert.

The heat-producing system presents the chief novelty. It was made up of two parts, a bottle (*X*) and a burette (*Z*). The former was made of platinum, with a capacity of 0.17 liter and weighing 52.64 grams. In this was placed a definite weight of a somewhat dilute, exactly known solution of sulphuric acid. The liquid was agitated by the platinum stirrer (*J*), alternating 145 times per minute. The bottle rested upon the glass triangle (*N*), thus permitting a free circulation of the calorimeter liquid around the entire surface. Tightly fastened into the neck by a small rubber stopper was the tip of the burette (*Z*), which contained a concentrated solution of soda. The discharge of this solution into the acid, and the consequent heat evolved by the reaction, formed the heat-producing action upon which the method is based. Since the alkali was the only part of the reacting system which, from its position, might, at the beginning of an experiment, have a different temperature than that of the remainder of the system, one needed to measure its temperature accurately. To this end the thermometer (*K*)[21] was immersed in the liquid, in which the stirrer (*O*) oscillated 145 times per minute. Concentric layers of heavy white silk aided in protecting the liquid mass from outside fluctuations of temperature. The drainings which collected in the lower end of the delivery tube after the admission of the soda to the bottle were expelled by blowing with a rubber bulb through the side tube (*H*).

It is of the utmost importance that the stirring should be efficient. The entire system of stirrers was driven by a small electric motor, a system of wooden pulleys giving the required reductions in speed. The stirrers of the bottle, jacket, and burette formed one system, and those of the calorimeter and cover, a second. It was found advantageous to attach the various oscillating stirrers to metal rods working in sleeves and actuated by cords fastened eccentrically to the proper pulleys. In this way uniformity of travel and stroke were secured, the friction of the rods in the sleeves being reduced by good lubrication to a negligible quantity.

As uniformity of composition in the acid used in the bottle within the inner vessel of the calorimeter is a fundamental condition for the accuracy of the process, the familiar device shown diagrammatically in Figure 2 was used for delivering it. The acid was stored in the 2-litre Jena flask (*A*) closed with a perforated rubber stopper. Through the siphon (*S*) the acid could be drawn into the burette (*B*). The auxiliary tube (*T*) equalized the pressure in the two containers. After

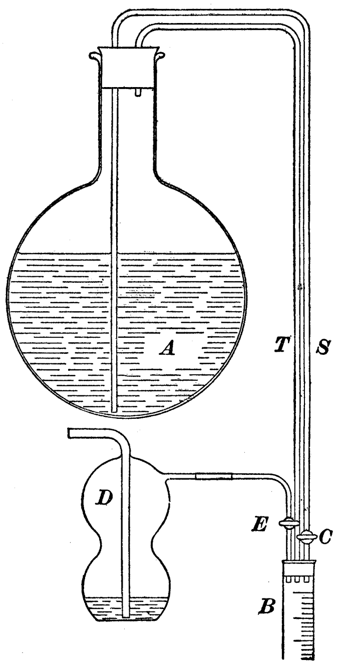

FIGURE 2.

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[21] The thermometer was a very accurate one, made especially for this purpose. It has a range of but 8 degrees, graduated in tenths.

filling the burette, the inflow cock (*C*) was closed. When the acid was to be drawn from the burette, the cock (*E*) connecting with the outside air through the wash bottle (*D*) was opened and the pressure thus equalized. As the wash bottle was filled with acid of the same concentration as that in the reservoir, the tension of aqueous vapor of the air introduced was the same as that obtaining in the system. The flask was always shaken before anything was drawn from it. By this means an acid was secured of unvarying composition, as shown by numerous experiments. In a similar way, with the addition of a soda-lime tower for the removal of carbon dioxide, the alkaline solution was maintained at constant strength.

It is needless to say that the thermometers were compared with Sevres standards with the greatest care, especially that designated *M*. Successive standardizations at different times were gratifyingly concordant.

Conduct of an Experiment.

The calorimeter proper (*W*, Figure 1) was partly filled with about 0.47 litre of the desired liquid, — either pure water to serve as a standard, or a solution to be studied. It was then brought to the temperature selected for the experiment, accurately weighed, and placed inside the jacket (*E*, Figure 1). This latter contained its charge of dilute crude alkaline solution, and was also near the selected initial temperature. About 0.1 litre of pure acid (1.34 normal) was then run into the platinum bottle (*X*, Figure 1), weighed carefully, and placed in a thermostat to bring it to the desired temperature. The innermost short burette (*Z*) was filled to the mark with about 0.02 litre of pure alkaline solution and brought near the required temperature. The whole apparatus was then rapidly assembled in the form already described. A few minutes after the stirrers were put in operation, the whole system was in thermal equilibrium, as was shown by the constant readings of the various thermometers. The temperatures of the calorimeter and the pure alkaline solution, indicated by the thermometers *M* and *K* respectively, were then carefully recorded, the stirrer in the bottle was disconnected, and the pure alkali discharged into the bottle as rapidly as possible. The immediate temperature rise, as the heavy alkali sank through the acid, was paralleled outside by running acid into jacket and cover. The bottle-stirrer (*J*) was then agitated by hand, this permitting excellent control of the mixing of pure acid and alkali and the resulting rise in temperature. When the mixing was almost complete, as shown by the rise of the thermometer *M*, the stirrer was reconnected with the motor and the final mixing done mechanically. The changes in the calorimeter throughout the experiment were carefully duplicated in the jacket and cover. At the end of some nine minutes the final equilibrium was attained, the thermometer readings becoming constant, at a temperature about four degrees above the initial temperature.

The calculation was exceedingly simple except for two features, each of which concerned the sodic hydroxide. The first of these was a correction needed because the alkaline solution had not exactly the temperature of the calorimeter at the moment of delivery. If warmer, the alkali brought with it a slight excess of heat; if cooler, it caused a slight deficiency. This correction was easily calculated by multiplying the water equivalent of the alkaline solution by the difference of temperature. When the alkali was too warm, this small product was subtracted from the total; when too cold, added. The other unusual feature involved not the total amount of alkali, but only the *excess* of this solution over and above the constant amount (19.30 grams) needed to neutralize the acid. It was intended that the alkaline solution should be of such concentration as to evolve enough heat on dilution to *raise itself* through the range of temperature of the experiment. If this were the case, it would not be necessary to know very exactly the amount of the alkali; any excess would not affect the final temperature. The alkali was made up as nearly as was possible on the basis of the previously known data to accomplish this result, and was nearly enough so for the present purpose. Its concentration was 8.97 normal.

The data and calculation of a specimen experiment may now be given without further preamble.

Specimen Experiment with Water in Calorimeter.

No. 4, February 27, 1908.

Data concerning temperature:

Initial temperature of calorimeter	16.489°
Final temperature of calorimeter	20.237°
Rise of temperature during experiment . . .	3.748°
Temperature of sodic hydroxide	16.44°
Difference between this and initial temperature	0.05°

Data concerning heat capacity, expressed in terms of the water-equivalent:

Water in calorimeter	474.97 grm.
Calorimeter and fittings, equivalent to .	11.35 "
103.71 grm. of dilute acid (sp. ht. = 0.94) .	97.49 "
19.3 grm. alkaline solution needed to neutralize acid (sp. ht. = 0.84)	16.21 "
Total heat capacity	600.02 grm.

Total heat observed = 600.02 × 3.748°	2248.87 cal. (18°)
Correction for heat needed to warm 20.9 grm. alkaline solution through 0.05° . .	+0.88 "
Total heat, corrected, from neutralization of 103.71 *grams acid*	2249.75 cal.
Heat evolved from 100 *grm. dilute acid* = $\frac{2249.75}{1,0371}$	2169.3 cal.

This process was repeated until there seemed to be no doubt as to the exact amount of heat evolved by the heat of neutralization of exactly 100 grams of this particular dilute acid by a slight excess of this particular alkaline solution under these perfectly definite conditions. The data and results of a series follow.

In the following table, T_1 is the initial temperature of the system and $T_2 - T_1$ is the observed rise. The other values are self-explanatory.

Results with Water.

No.	H_2SO_4	Total Water Value.	NaOH Correction.	T_1.	$T_2 - T_1$.	Corrected Heat.	Heat per 100 grams.
	grams.	grams.	calories.	Centigrade.	Centigrade.	calories (18°)	calories (18°)
4a	103.74	597.19	−4.74	16.03°	3.776°	2250.3	2169.1
5a	103.74	597.19	−3.51	16.37°	3.774°	2250.3	2169.1
2	103.69	600.02	+7.02	16.26°	3.736°	2248.7	2168.7
3	103.68	599.98	+5.44	16.37°	3.740°	2249.4	2169.5
4	103.71	600.02	+0.88	16.49°	3.748°	2249.8	2169.3
					Mean		2169.14

The maximum variation from the mean here is only 8 parts in 22,000, or about 0.02 per cent. As will be seen upon inspection, the correction for the difference in temperature of the alkali is sometimes additive and sometimes subtractive in the different experiments, hence the concordance of the observed results in connection with these values is excellent testimony as to the accuracy of the correction.

The amount of heat evolved by the neutralization of 100 grams of sulphuric acid under these conditions was now used as the standard in warming various definite solutions through about the same range of temperature. In order to accomplish this purpose, the solutions were successively placed in the calorimeter, and the flask for conducting the heat-producing neutralization was immersed in each just as it had previously been immersed in the pure water.

As an example, a series of results with a special solution of hydrochloric acid may be given. This acid was chosen for determination because, being involved in another research, its specific heat was a matter of immediate interest.

Below are given the data and method of calculating a single experiment, as well as the data of a series.

Specimen Experiment with a Solution.

No. 3, May, 1908.

Weight of dilute sulphuric acid in platinum bottle	103.72 grm.
Data concerning temperature:	
Initial temperature	16.236°
Final temperature	19.960°
Temperature rise	3.724°
Temperature of alkali	16.13°
Excess over initial temperature	–0.11°
Heat, producing this effect:	
Calculated heat evolved by reaction = 103.72 × 2169.14	2249.83 cal. (18°)
Heat taken by alkali = 20.9 × 0.84 × 0.11	–1.93 "
Total heat actually available in process	2247.90 cal.
Data concerning heat capacity, in terms of water equivalent: [22]	
Water value of calorimeter	10.87 grm.
Water value of acid	97.50 "
Water value of alkali	16.21 "
Total	124.58 grm.
Heat used by system exclusive of solution = 124.58 × 3.724°	= 463.94 cal. (18°)
Heat needed to raise 488.35 *grams of hydrochloric acid contained in calorimeter* = 2247.90 – 463.94	= 1783.96 (18°)

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[22] As these amounts are constant in all the determinations, slight constant errors in them would have only a vanishingly small pernicious effect upon the final results. The method is a *comparative* one, and small errors of this kind cancel out.

Hence, specific heat of hydrochloric acid of concentration $HCl\ 200.0\ H_2O = \frac{1784.0}{488.35 \times 3.724} = 0.9809$

The experimental data for this series are found in the accompanying table. Several experiments where the manipulation was faulty were rejected, but if they had been included the average would have remained essentially unchanged.

The Specific Heat of $HCl\ 200\ H_2O$.

No.	HCl.	H_2SO_4.	T_{NaOH}.	T_1.	$T_2 - T_1$.	Correction NaOH.	Specific Heat.
	grams.	grams.				calories.	
5	488.31	103.71	16.38°	16.39°	3.727°	0.00	0.9810
6	488.25	103.68	16.14	15.99	3.732	+2.63	0.9806
8	488.35	103.72	16.13	16.24	3.724	−1.93	0.9809
9	488.34	103.70	16.30	16.40	3.723	−1.76	0.9812
				Mean	. . . .		0.9809[23]

As will be seen, the maximum variation from the mean is 0.03 per cent. This experimental error is as low as could possibly be expected.

Heat of Dilution.

It is obvious that this apparatus can be applied to the accurate determination of the heat of dilution of any solution put into the burette (*Z*), if water instead of sulphuric acid is placed in the platinum flask (*X*). The liquid to be diluted is run into the bottle as before, and there mixes with a weighed amount of pure water. A series of three experiments on the dilution of a concentrated solution of sodic hydroxide is given below. The results are calculated in kilojoules, as the best standard for permanent record; in the experiments previously recorded this was unnecessary because the method was a comparative one and the dimension of heat energy was eliminated in the result. 0.100 litre of pure water was contained in the platinum bottle.

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[23] The corresponding values obtained from the results of Thomsen (loc. cit.) and Marignac (loc. cit.) are respectively 0.979 and 0.983.

THE HEAT OF DILUTION OF SODIC HYDROXIDE $NaOH \cdot 5.85 H_2O$.

No.	Water Value.	NaOH.	$T_2 - T_1$.	Corr. (NaOH).	Heat evolved by Dilution to $NaOH \cdot 43.5 H_2O$.
	grams.	grams.	° C.	calories.	kilojoules.
1	601.79	21.39	0.132	– 4.5	3.82
2	601.86	21.43	0.129	– 3.2	3.78
3	602.01	21.59	0.155	–18.5	3.77
			Mean		3.79

The variation from the mean falls within the probable experimental error (0.001°).

Obviously any thermochemical effect produced by the mixing of two liquids could be measured in the same way. It is to be noted that the method has a great advantage over other methods in that great speed in the execution of the experiment is not at all necessary. By the old methods, speed was essential because of the correction for cooling; but here there is no correction for cooling because the performance is strictly adiabatic. The reaction may extend over hours, if necessary.

It should be noted that the correction concerning the sodic hydroxide could be wholly avoided if the pure alkaline liquid were contained in a receptacle within the calorimeter, instead of being held in a burette above it. Such a receptacle has been used successfully by Richards and Henderson [24] and was not introduced in these preliminary experiments on account of its slightly greater complexity. In the future it will be adopted, and with it we hope to secure yet more accurate results.

Experiments are now under way for the determination of the specific heats and heats of dilution of various solutions at different concentrations and at different temperatures, by the methods just described.

It is a pleasure to acknowledge the generous aid of the Carnegie Institution of Washington, without which we should have been greatly hampered in this work. The present and future results of this investigation will be published in greater detail by that Institution, in one of its shortly forthcoming regular publications.

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[24] These Proceedings **41**, 11 (1905); Zeit. phys. Chem., **52**, 551 (1905).

Summary.

The results of this paper may be briefly summarized as follows:

1. A new method for the accurate determination of the specific heats of liquids has been described, using the adiabatic calorimeter and a chemical source of heat.
2. The heat capacity of a solution of hydrochloric acid of molal concentration $HCl + 200\ H_2O$ has been measured.
3. The method has been applied to the accurate determination of heats of dilution.
4. A solution of alkali was used whose heat of dilution automatically compensates for any excess which might have been added.

Chemical Laboratory of Harvard
College, April 27, 1908.